Laboratory trials on desiccant dust insecticides

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Abstract

Laboratory comparisons of six desiccant dust insecticides were made to estimate their suitability for the control of insects in grain, grain stores and silos. Efficacy tests for structural treatment used surface applications of dust and aqueous slurries. Materials tested included silica aerogels and modified diatomaceous earths.

Grain treatment results indicate that: a) valid assessment of this type of insecticide requires longer test exposures than those generally used for chemical toxicants: b) there is, in general, an inverse relationship between particle size and efficacy with desiccant dusts: c) most of the products tested would protect grain within the range of concentrations used in these trials: d) minimum concentrations required to effect long-term insect control estimated from the results corresponded reasonably well to those found in commercial practice: and e) despite their very different physical properties and efficacies, all six products tested produced mortality/exposure-time curves with common characteristics.

Surface treatment results showed the dried deposits obtained from slurries proved to be less effective than dust applications and only two of the test products, one aerogel, Dri-die®, and one modified diatomaceous earth, Dryacide®, performed adequately as surface treatments in both dust and slurry form.

Introduction

There is an acknowledged interest in replacing toxic chemical insecticides with less hazardous technologies for stored grain protection. Concern about the increasing numbers of chemical-resistant strains of stored grain insect pests, adverse environmental impacts and a growing demand for 'chemical-free' food products are aspects of this problem. Desiccant dusts offer an acceptable alternative to the use of chemical insecticides in many grain storage situations.

These products are based on inert minerals such as silica gel or diatomaceous earth and contain no chemical insecticide or knock-down agents. They are also residue-free, effective against chemical-resistant species, and typically very persistent. Most products, at the appropriate concentration, provide protection for at least 12 months. They are stable at high and low temperatures and are non-selective for resistance.

Various studies on the efficacy of desiccant dusts against stored-grain insect pests have been reported (White et al. 1966; La Hue 1970; Patourel 1986; Desmarchelier and Dines 1987; Aldryhim 1990, 1993). However, products tested were limited and variations in test conditions, insect species and exposure periods make it difficult to compare efficacies of the several products on the market.

In recent years, considerable Australian research has been carried out on desiccant dusts for the structural treatment of grain stores and grain-handling machinery. This method has received general acceptance by Australian grain industries over the past three years and is contributing to the residue-free quality of much of Australia's grain. It can also offer considerable cost savings over chemical treatments and provide more effective, long-term protection (Desmarchelier et al. 1994).

The recent interest in aqueous slurry applications of desiccant dusts reflects the preference for clean application methods with minimal operator exposure to dusts. However, the benefits are not restricted to meeting low-dust operating conditions demanded by employers and trade unions. The dried slurry deposit is clearly visible on treated surfaces and this permits improved control of coverage and application rates. The speed of application is also significantly higher than that obtained when applying chemical insecticides. Many Australian grain industry operators have now switched from dust to slurry applications.

Materials and Methods

Test species

Adult granary weevil (*Sitophilus granarius*) was chosen as the test insect as this species has been identified by CSIRO Stored Grain Research Laboratory (SGRL) as one of the most difficult stored-product insect pests to control with desiccant dust formulations. In the trials involving surface exposures to dusts and slurry-deposited films, the rice weevil (*Sitophilus oryzae*) was also included as an additional test insect. All insects used were drawn from laboratory cultures which had been maintained over several years.

Grain treatment

The required quantity of dust was weighed into a 160 mL glass jar to which was added 40 g of wheat. The dust was mixed into the wheat by simultaneously rotating and inverting the jar for 15 seconds, shaken vigorously for 5 seconds, then again rotated and inverted for a further 30 seconds. Forty adult insects, collected within 7 and 21 days of emergence, were used in each jar. As the insect colonies contained insufficient numbers to provide a common source for the entire series, different colonies were used to provide four separate replicates. Each colony provided sufficient insects to cover all product-concentration combinations for the full 21-day series.

Bioassays were carried out on wheat with moisture content of 12% and stabilised for at least 4 weeks in a sealed container before use. Pre-trial bioassays using very wide ranges of treatment rates were carried out to establish five or six treatment concentrations for each product which would yield mortalities in a 10–99% range during the test period.

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Samples were retained in a constant temperature-humidity room (25°C, 56% r.h.), for the exposure period. These test conditions reflect the typical microclimate prevailing in wheat stored at the maximum moisture content accepted by bulk grain handling authorities in Australia. They therefore reflect moderately adverse conditions for testing desiccant dust insecticides.

As it was evident from earlier trials that frequent assessments were necessary to establish the time-mortality response of the different products, assessments were made every 2 days until the 18th day with a final count on the 21st day. The data were analysed using log-dose-probit mortality regressions.

Surface treatment

The laboratory test protocol employed was developed by SGRL. It is considered to provide a reasonable guide to the potential of dust formulations for this end use (Desmarchelier and Allen 1988). Since one of the products tested, Dryacide®, has been successfully used by Australian grain handlers for structural treatment in both dust and slurry applications, it also provided a benchmark against which other products could be compared.

Slurry application

Aluminium baking trays (272 × 212 × 60 mm) were degreased and cleaned, then scoured very lightly with a steel-wool pad to provide a slight roughening to the inside surface. A fine spray of 20% w/w slurry was applied to the tray base and vertical sides by running a finger across the bristles of a small scrubbing brush (70 × 30 mm with 20 mm bristles) which had been dipped into the slurry then shaken to remove all excess. The tray was oven dried at 50°C after each application and weighed. Multiple, light applications were made until the desired dry weight (580 mg), corresponding to 5 g/m^2, had been achieved. Consistent and uniform applications could be achieved in this way and the resulting dried deposit reflected very closely the spray deposit achieved by high pressure spraying used commercially in Australia. Extensive laboratory and field trials (Desmarchelier and Allen 1988) have demonstrated that varying surface application rates between 5 and 9 g/m^2 (dry basis) make little difference to the efficacy of one of the dusts tested, Dryacide®, and the 5 g/m^2 corresponds closely enough to the Australian registered application rate for that product, namely 6 g/m^2.

Insects were prevented from escaping by closing the trays with aluminium lids crimp-closed inside the tray edge. The seals did not prevent air exchange and the internal environment would rapidly equilibrate with the test room conditions.

Dust application

Aluminium trays (272 × 212 mm) were cleaned and prepared as previously detailed then placed on a digital balance. Dust was applied through a gently shaken 150 µm screen to give an even coating to the base, as determined visually, until the desired 120 mg application weight, corresponding to 2 g/m^2, was achieved. The application rate of 2 g/m^2 was adopted to correspond to the Australian registered treatment rate for Dryacide®, the only desiccant dust registered in Australia.

Test conditions

Eighty adults of the species under test were placed inside the trays which were retained in a constant temperature/humidity room (25°C, 56% r.h.) for the desired exposure periods of 26 hours (rice weevil) and 30 hours (granary weevil). Three replicate trays were employed for each trial, each replicate being sourced from a different colony. On completion of the exposure time, surviving insects were placed on clean, residue-free wheat, 12% m.c., and retained in the same constant temperature-humidity cabinet for a further 6 days.

Mortalities were assessed immediately after the exposure period (initial), after one day on clean wheat (acute), and after the 6th day (end-point). Extensive use of this test protocol by the author had confirmed that mortalities after the 6th recovery day were very low and could be discounted. Exposure periods were chosen to maximise discrimination between products. When exposures of 48 hours or longer are adopted, starvation makes an increasingly larger contribution to mortality, as is shown by the controls. The exposure periods adopted in these tests resulted in negligible control mortality.

Test products

Only commercially available dusts were selected, all of which were found to be based on amorphous silica. Although investigations on alternative materials have been reported, e.g. activated kaolin (Permual and Patourel 1990) and magnesite (Milvain 1992), these were not as effective as the commercially available alternatives.

Most of the dusts selected for testing were proprietary brands recommended for the control of stored-product insects: Insecto®, Dryacide®, Insectigone® and Perma-guard® are modified diatomaceous earth products. Other dusts were tested which are not specifically registered for the control of stored-grain insects. However, their efficacy against other insect species justified their consideration for this application. Dri-die is a precipitated silica aerogel incorporating 2% fluoride adjuvant and used to control insects including dry-wood termites and insect pests of domestic animals. Aerosil R974 is a hydrophobic fumed silica showing excellent insecticidal properties. A fine particle size grade of Dryacide®, Dryacide® UF, was also included to evaluate the effects of particle size, and therefore surface area, on efficacy. This sample was separated from commercially available Dryacide® by air classification, only the fine fraction being retained for the sample. Physical characteristics and supplier details of products tested are given in Appendix 1.

Results

Grain treatment

The cumulative mortality responses to exposure time for three of the test products are shown in Figure 1. The products were chosen to illustrate typical responses of an effective silica gel, Aerosil R974®, and the most and least effective of the diatomaceous earth products, Dryacide® and Perma-guard®. The response curve shown for Aerosil R974® was typical of both silica products at all concentrations tested. Typical sigmoid mortality-response curves were shown by Dryacide® and Perma-guard® and this shape typified the response of all the diatomaceous earth products. However, an assumption that the different response pattern reflects a fundamental difference in mode of action could not be sustained. Both Dryacide® UF and Dryacide® yielded response curves similar to those of the aerogels at high dust concentrations. This type of response is evidently a truncated sigmoid curve.

More importantly, Figure 1 clearly illustrates that there is no single 'correct' exposure period for the assessment of efficacy of desiccant dusts.

Log_{10} concentration (g/t)-probit response curves were calculated for each sampling time. No corrections were made for control mortality as in no case did this exceed 2% despite repeated mortality estimates at 2-day intervals. Figures 2 and

3 show LC_{50} and LC_{90} values for the test dusts at each sampling interval during the total period of exposure. In both graphs, only interpolated values have been included to maximise reliability: i.e. trials in which no concentration achieved 50% mortality, or those for which all concentrations produced mortalities exceeding 50%, have been excluded from the LC_{50} plots.

The results illustrate the very strong dependence of mortality on duration of exposure. Clearly the standard 96 hour LC_{50}, which nominally samples mortality after the 'acute' phase is completed, would not represent an appropriate time to evaluate the potency of desiccant dusts under these experimental conditions. The slopes of the response lines, particularly the diatomaceous earths, indicate that mortalities of 50% and 90% would have been obtained at even lower desiccant concentrations if the experiment had been continued.

The two aerogels, Aerosil R974® and Dri-die®, were clearly faster-acting and more potent insecticides in the short term. However, in both cases, the mortality curves flattened considerably after the first weeks exposure, and Figure 3 shows that after 18 days exposure the LC_{90} for Dri-die® is greater than that of Dryacide® UF which now has a higher potency. The curve of the latter product also shows some indication of flattening out after this interval and unreported trials on Dryacide® by the author have confirmed that the same pattern does apply to that product at lower concentrations over longer periods. It is likely to be a common response pattern of all desiccant dusts.

From these data it is impossible to make a judgement on efficacy without simultaneously considering the duration of the exposure. Nevertheless, the results shown in Figures 2 and 3 are highly consistent. Aerosil R974® shows the greatest potency over the experimental period. The curves of Dryacide® UF and Dri-die® overlap, but the other products maintain a consistent relationship, with Perma-guard® having the lowest potency and Dryacide® the highest potency of the remaining diatomaceous earth products.

Many published reports on the efficacy of desiccant dusts have relied upon mortalities achieved for a single, usually short, exposure period. The slope of the probit-log dose regression line, *S*, is then estimated from the single response curve to estimate LC_{50}, LC_{90} or other relevant concentrations. In the test series reported here, 10 estimates of the slope, *S*, were made at 2-day intervals over the 21-day test period.

Initially the slopes were quite variable, but after 8–10 days they remained reasonably constant for the remaining period of exposure. By averaging the slopes of the final 5 or 6 trials, more precise and reliable estimates of the mortality responses to changes in dust concentration could be made. Twenty-one day 'end-point' LC_{50} and LC_{90} values were estimated in this way and the results are shown in Figure 4. Quarles (1992) suggested that this is the minimal exposure period which should be used for mortality estimates when insects are on grain.

It is worth noting, however, that after 21 days exposure Dryacide® UF is clearly more effective than both Dri-die® and Dryacide®. Figure 4 shows that most of the relative potencies of the test products remain consistent with those described earlier. This is a clear demonstration that, for some desiccants at least, higher potencies can be achieved simply by using a smaller particle size. Dryacide® UF is simply the 'fines' separated from a standard batch of Dryacide® by air classification.

Surface treatment

The results of surface treatment are shown in Figures 5 and 6. The greater susceptibility of *S. oryzae* to desiccant dusts is evident in the slurry exposure trials, though not so in the dust exposure tests. This is contrary to the results observed in previous grain-treatment trials carried out by the author and may be a consequence of the shorter exposure time.

Dust application

The two aerogels and Dryacide® UF gave virtually 100% kill by the 6 day 'end-point' for both species. The overall responses showed a similar pattern of efficacy to those seen with the grain treatments.

Slurry application

When applied as an aqueous slurry, few of the products were effective, particularly against the granary weevil (Fig. 6). This is probably a result of the lower pick-up rate from dried slurry deposits and was definitely the cause of the loss of efficacy demonstrated by Insectigone®. The dried residue of this product bonded strongly to the substrate. However, such was not the case for Aerosil R974®. This strongly hydrophobic dust required the addition of a non-ionic detergent (0.5% w/w) and dispersion in an intensive laboratory mixer to 'wet' it and obtain a slurry. The dried deposit 'dusted' and transferred to the test insects at roughly the same level as Dri-die®, but its efficacy was substantially inferior to that obtained when it was deposited as a dust. From the results obtained in these trials, only Dri-die® and Dryacide® gave efficacies which might justify applying them in slurry form.

Discussion

Grain protection

The results of these trials illustrate the problem of assessing the efficacies of desiccant dusts: they are not directly toxic, are relatively slow acting and typically become increasingly effective with longer exposure times. Since the principal mode of action appears to be as a desiccant, products are sensitive to the moisture content of grain (Desmarchelier and Dines 1987; Patourel 1986; Aldryhim 1990, 1993) or ambient humidity when used in structural treatment applications (Milvain 1993).

Because effective insect control in commercial grain stores requires both adult insect eradication and progeny control in conditions which may vary quite significantly over the storage period, only field trials can determine real product efficacy. Such trials should be long-term, be conducted in relatively adverse conditions of moisture content, ambient humidity, and use resistant insect species.

One of the products used in the current trials, Dryacide®, has been in regular commercial use in Australia for on-farm stored grain protection since 1985. Most of such grain has been treated and stored at or below the 12% moisture content limit stipulated by State grain authorities. The treatment rate in all cases is 1000 g/t. The results of monitored field trials and commercial use on farm-stored wheat, oats and barley, in Australia have demonstrated Dryacide®'s ability at the recommended dose rate to eradicate all but heavy infestations and prevent re-infestation by invading insects over extended periods of time (Swarts 1980; Arthur 1980). Emerging progeny have also been controlled. It should therefore be possible to relate the results of these laboratory efficacy trials with actual field performance.

The laboratory trials showed that virtually the same 1000 g/t concentration of Dryacide® (982 g/t test result) gave 99% mortality on the 21st day and, by extrapolation of the log dose-

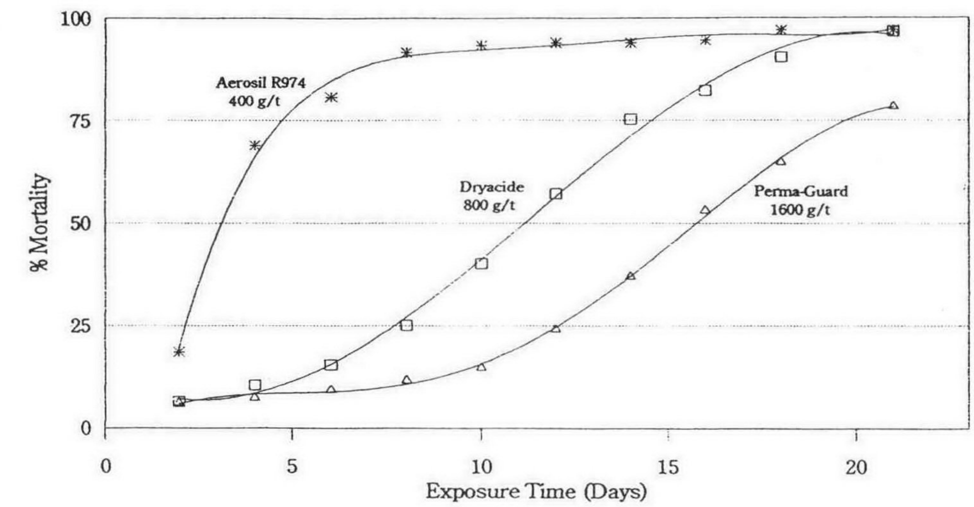

Fig. 1. Cumulative mortality of granary weevils (*Sitophilus granarius*) exposed to wheat treated with three desiccant dust insecticides.

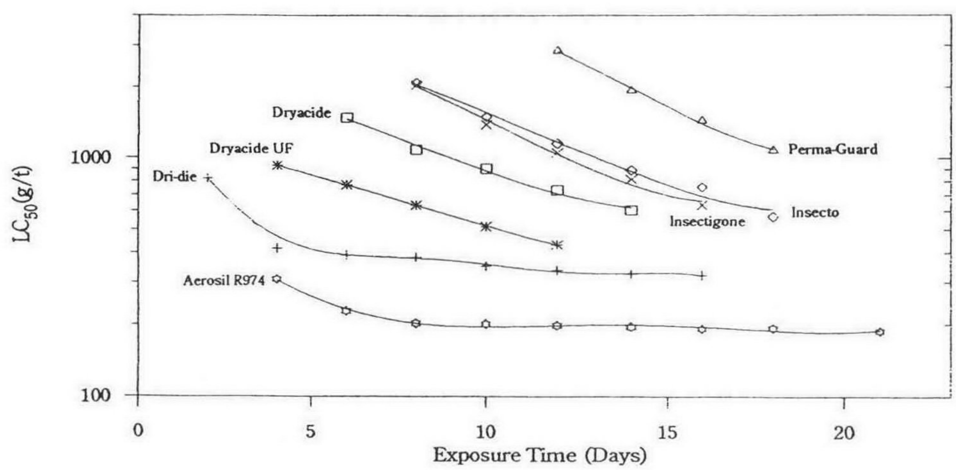

Fig. 2. Relationship between LC_{50} of granary weevil (*Sitophilus granarius*) on wheat treated with various desiccant dusts and duration of exposure.

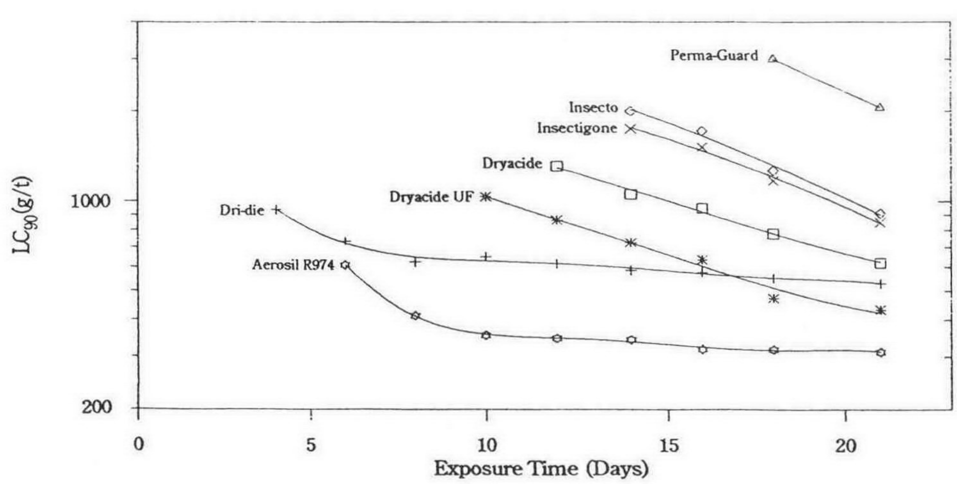

Fig. 3. Relationship between LC_{90} of granary weevil (*Sitophilus granarius*) on wheat treated with various desiccant dusts and duration of exposure.

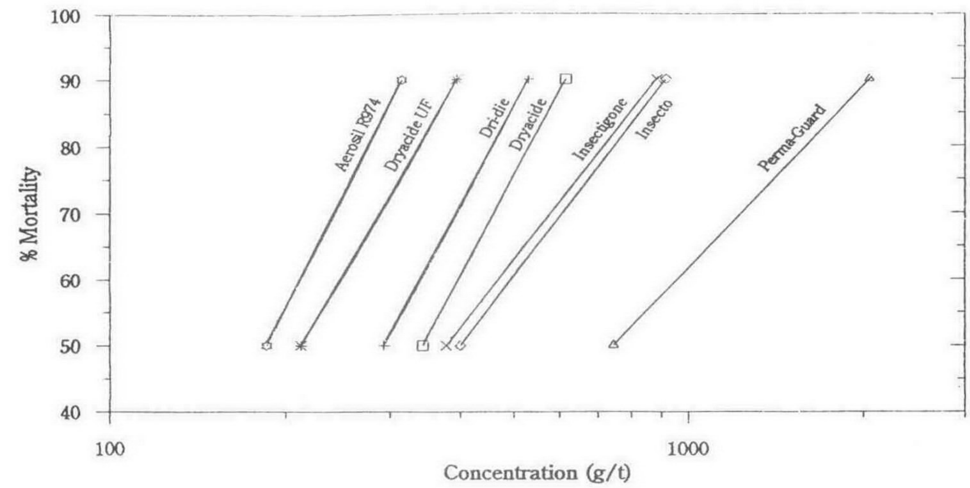

Fig. 4. LC_{50} and LC_{90} of the granary weevil (*Sitophilus granarius*) on wheat treated with various desiccant dusts at an exposure time of 21 days.

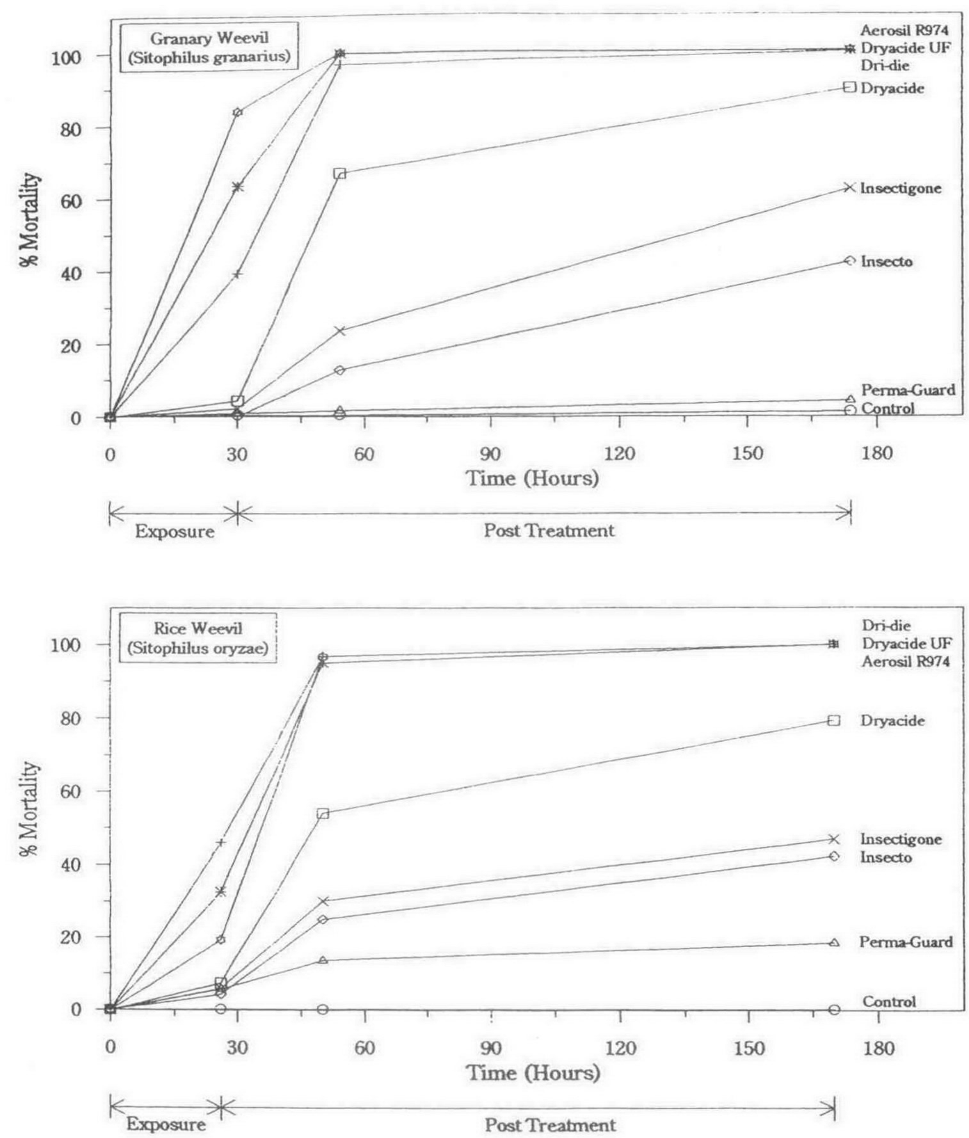

Fig. 5. Cumulative mortality of grain weevils following exposure to surfaces treated with inert dusts (2 g/m^2).

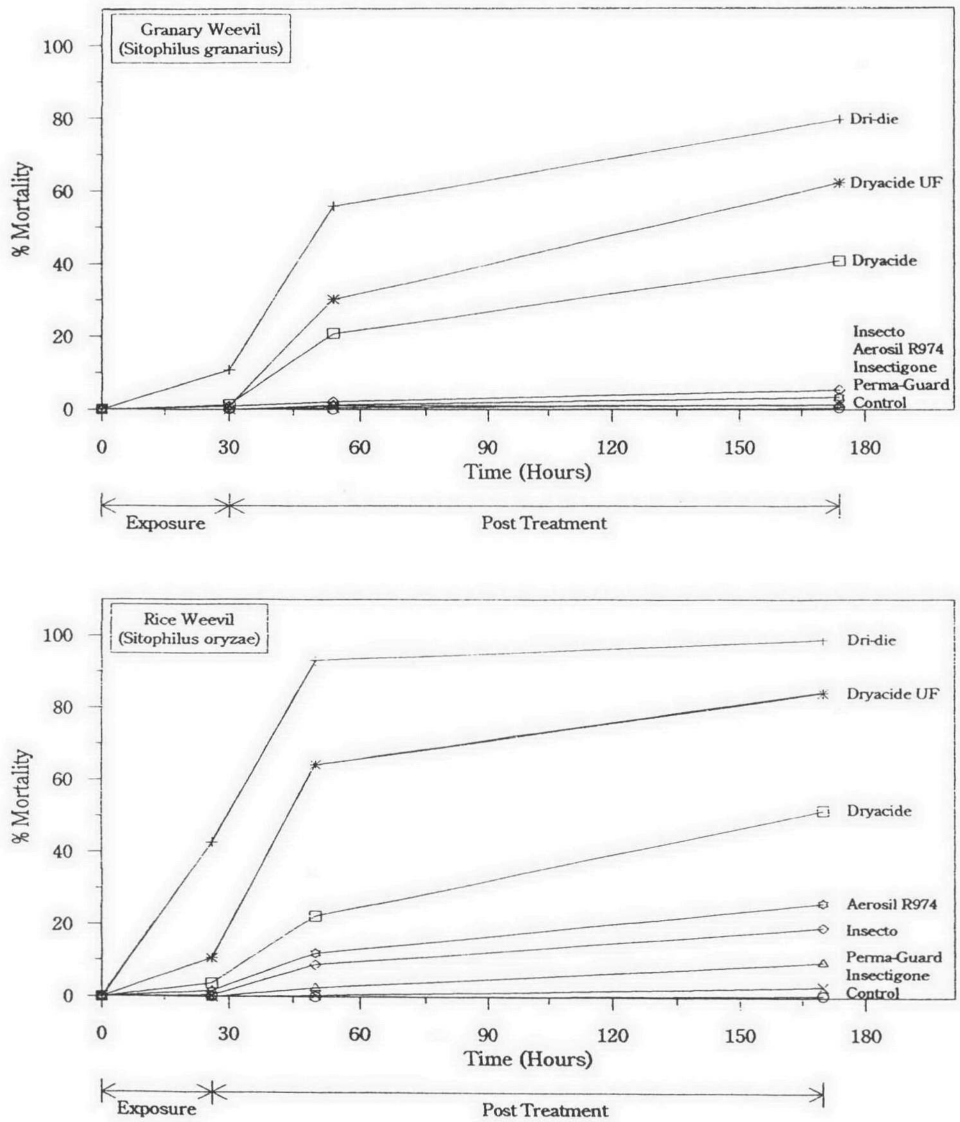

Fig. 6. Cumulative mortality of grain weevils following exposure to surfaces treated with inert dusts applied as aqueous slurries (5 g/m^2).

probit curve, would give an estimated 99.99% mortality on the 28th day. This can be related to the test protocol developed for desiccant dust preparations by SGRL and the Western Australian Department of Agriculture, which stipulates 100% mortality of 100 test insects within 28 days, the test species and conditions being identical to those used in this trial. If the achievement of 99% mortality within 21 days, using the test protocol employed in this series of tests, were adopted as a criterion for suitability and to estimate control concentration rates, Aerosil R974®, Dri-die®, Dryacide® UF and Dryacide® would meet it with estimated concentrations of 470, 860, 640 and 980 g/t, respectively. The corresponding dosage rates for Insectigone®, Insecto® and Perma-guard® would be: 1910, 1830 and 4760 g/t, respectively.

The results of long-term field trials in the USA on grain protection afforded by silica aerogels and diatomaceous earth products have also been reported (Quinlan and Berndt 1966; La Hue and Fifield 1967; Strong and Sbur 1963; White et al. 1966; La Hue 1970). Although there is some conflicting evidence on relative efficacies achieved, a consistent conclusion arising from their results is that 12 months protection in typical USA grain storage conditions is provided by diatomaceous earth products at a dosage rate of 3500 g/t. The repellent effect on insects exposed to grain treated with high dosage rates of diatomaceous earth was considered to be a contributing factor in this long-term control.

To provide the same long-term protection, silica aerogels such as Cab-O-Sil® and SG68 (Dri-die without the 2% fluoride adjuvant) required treatment levels of 750 to 1000 g/t. Thus, the dosage rates of Dryacide®, aerogels and diatomaceous-earth-based products demonstrated as being necessary to achieve control in commercial practice or field trials correspond broadly to those rates indicated by this series of laboratory tests.

Structural treatment

Of the products tested, only Dryacide® has been in regular commercial use for structural treatment of grain storage facilities, with both dust and slurry treatment methods being employed. As this was an innovative development in Australia, its performance has been monitored by researchers from CSIRO and State Bulk Grain Handling Authorities over several years. Investigations reported by Desmarchelier et al. (1994) and Bridgeman (these proceedings) have confirmed the ability of Dryacide® dust and slurry deposits to maintain effective, long-term (12 months or more) control of stored grain insect pests.

Dryacide®'s performance in the laboratory trials reported here can be used as a standard against which the short-term effectiveness of the other products can be measured. Longer term efficacy of the other products cannot be assessed without appropriate trials as there is insufficient information on substrate bonding and stability of their dry and slurry-applied dust films. Too strong a bond can result in insufficient dust pick-up and low efficacy, as was the case with Insectigone. Bond strengths which are too low may result in total loss of the deposit over a relatively short period of time. As grain stores, silos and grain handling machinery are frequently out of use for many months between harvests, maintained efficacy, particularly with tolerance of temperature extremes, is a highly desirable feature of structural treatment insecticides.

One clear conclusion from these trials is the importance of particle size in determining efficacy of insecticidal dusts. This is particularly the case in dust applications, and the strong performance of the two aerogels supports this assessment. The enhanced effectiveness shown by Dryacide® UF, which differs only from the standard product in particle size distribution, in both dust and slurry applications, also reinforces the conclusion drawn by other investigators (Melichar and Willomitzer, as cited by Ebeling 1971; Parkin 1974), that efficacy is strongly influenced by the particle size, and therefore surface area of the preparation.

Efficacy is not the only factor affected by particle size. Despite the classification of these amorphous silica dusts as inert, or nuisance dusts with Occupational Environment Exposure Standards of 10 mg/m^3, the particle sizes of Dryacide® UF, Dri-die® and Aerosil R974® place these products in the range of respirable dusts and therefore represent a potentially greater health hazard to operators and end-users than the other products tested.

Role of desiccant dusts in the grain industry

A drawback to the use of desiccant dusts for grain protection is the very high treatment rates required and the resulting effect on the physical properties of the grain. These effects prohibit the use of desiccant dusts for much of the bulk grain stored and exported round the world at the present time. However, this drawback may become of less importance when solutions are being sought to the problems of residue contamination of food commodities, the emergence of resistant insect strains and the need to maintain long-term control, even with extremes of storage temperatures. One recent example of such an attitude change was the adoption of a desiccant dust to protect stored paddy rice and conform to 'organically grown' produce requirements (R. Corner, pers. comm. 1994).

In small grain-storage facilities where grain handling properties have little if any significance, and when climatic and storage conditions are appropriate for the use of desiccant dusts, they offer a safe and reliable alternative to chemical insecticides.

There will probably be considerable expansion in the use of desiccant dusts for fabric treatment of storage and transport facilities and grain handling machinery, particularly in slurry form due to ease and cleanliness of application, visibility and cost effectiveness. In Australia, this application in extending beyond grain storage into the protection of food-processing facilities and food-product warehouses.

New developments

One new application of the desiccant dust, Dryacide®, developed by Australian researchers is its use as a grain surface coating in SIROFLO® systems, a new fumigation technology developed by CSIRO (Winks 1994). The inert dust blanket, 100 g/m^2, acts as a gas membrane, retaining higher fumigant concentrations in the surface layers of grain. The insecticidal desiccant dust layer also provides an additional safety margin for insect control. The introduction of this new strategy in Australia's bulk grain storage systems has improved grain hygiene and extended the SIROFLO® technology into horizontal stores. The small amount of dust added does not affect grain physical properties or post-treatment handling. A related application is also being investigated in Australia. Grain-surface treatment with Dryacide® at the same 100 g/m^2 rate appears to improve insect control in bulk aerated grain stores (Nickson et al., these proceedings).

Acknowledgments

R. H. McLaughlin and A. Mostofi are thanked for their assistance in preparing the manuscript and Y. Hawkins for the literature survey. The author is grateful to J. M. Desmarchelier, S. E. Allen and their colleagues at SGRL for their invaluable advice and support.

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Appendix I : Desiccant Dust Products

Suppliers

Aerosil R974—Degussa AG, Weissfrauenstrasse 9, D-6000 Frankfurt 11, Germany. A hydrophobic fumed silica used as a powder flow improver and thickener in water-resistant systems such as paints and offset inks.

Dri-Die—Fairfield American Corporation, 201 Route 17 North, Rutherford, NJ 07070, USA. A silica aerogel containing 2% fluoride adjuvant and used to control dry-wood termites and insect pests of domestic animals.

Dryacide and Dryacide UF—Dryacide® Australia Pty Ltd, 1/20 Ryelane Street, Maddington 6109, Western Australia. A silica-gel modified diatomaceous earth desiccant dust insecticide.

Insectigone—Chemfree Environment Inc, 16763 Hymus Blvd, Kirkland, Quebec H9H 3L4, Canada. A modified diatomaceous earth desiccant dust insecticide.

Insecto— Insecto Products Inc., 630 North Eckhoff Street, Orange, California 92668, USA. A modified diatomaceous earth desiccant dust insecticide.

Perma-Guard FSF Grain Treatment—Perma-guard Inc. P.O. Box 25282, Albuquerque, New Mexico 87125, USA. A modified diatomaceous earth desiccant dust insecticide.

Physical properties of dessicant dust products.

Product	Mean particle size (μm)	Bulk density (g/L)	Surface area (g/m^2)	Composition (wt %)
Aerosil R974	0.012	50	170	99.8% SiO_2
Dri-die	<0.1	75	300	95% SiO_2
Dryacide®	13–15	300	3	90% SiO_2
Dryacide® UF	1.5	300	11	90% SiO_2
Insectigone	NA[a]	350	NA[a]	80% SiO_2
Insecto	7–8	350	3	90% DE[b]
Perma-guard	15–20	350	2.5	90% DE[b]

[a]Not available
[b]Diatomaceous earth